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INFLUENCE OF STRESS CORROSION ON
STRENGTH OF GLASS FIBERS

(Unclassified)

Third Bi-Monthly Progress Report

September 30, 1964

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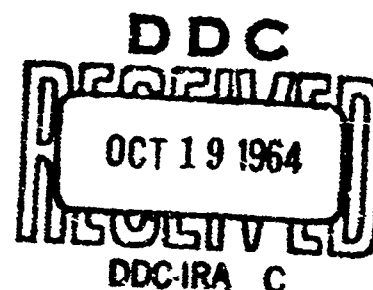
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Report submitted by:

Advanced Engine & Technology Dept.
General Electric Company
Evendale, Ohio



This work was performed under Contract No. Nonr 4486(00) (X)
for the U.S. Naval Research Laboratory.

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INFLUENCE OF STRESS CORROSION ON STRENGTH OF GLASS FIBERS

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I. SUMMARY

Initial static fatigue tests at liquid nitrogen temperature on single filaments of E-glass yielded incomplete data because of frost build-up problems. It became necessary to isolate the equipment from room humidity. This has been done and low temperature testing has resumed successfully, with delayed failure data accumulating at a satisfactory rate. The results so far indicate that even with the very high rates of loading used in these tests, the stress level at which most fibers will fail immediately is lower at room temperature and normal humidity than at liquid nitrogen temperature. Gradually raising the temperature from liquid nitrogen temperature causes the failure of fibers which have been supporting a given load at -196°C . The implication is that the processes involved in static fatigue are greatly slowed down, but not completely eliminated, by lower temperatures in the range from room temperature to -196°C . Complete static fatigue results at -196°C are not yet available.

II. INTRODUCTION

In the second bi-monthly progress report on this project, static fatigue test results at normal atmospheric conditions were reported. This study yielded three important conclusions:

- A. Static fatigue, or delayed failure, occurs in E-glass single filaments at all stress levels from 400,000 psi down to 200,000 psi. (No tests were run at stress levels lower than 200,000 psi).
- B. When fixed dead weight loads are applied rapidly, as they are in these static fatigue tests, the percentage of immediate failures decreases quite uniformly as the stress level is reduced.
- C. For those fibers which survive the initial application of load, the most probable failure time becomes longer as stress level is reduced.

These effects were discussed at some length in the previous report. One other observation from the room temperature tests deserves some comment. The apparent strength level of fibers loaded by the procedure employed in the static fatigue tests is much lower than that measured by the conventional tensile test carried out at known strain rates. For example, some

prior work under Navy contract Now 61-0641c (FBM) had shown that E-glass fibers produced in this laboratory had a median strength of 507,000 psi when measured in an Instron tensile test machine using a strain rate of 0.05 min.^{-1} . A representative sample of 10 fibers from the same lot used in the current static fatigue tests was found to have a median strength of 497,000 psi by the Instron test. In comparison, a load which produces a static stress of only 350,000 psi, when rapidly applied in our static fatigue experiments, caused 50% immediate failures at room temperature.

It has been a common observation by many investigators that strain rate has an appreciable effect on measured strength of glass fibers. Low strain rates have tended to yield poorer strengths, and this has been one factor lending support to the hypothesis that stress corrosion is responsible for strength loss in this material. Our much lower strengths under very rapid loading are therefore not to be explained by this mechanism.

It is our feeling that the rate of load application in our static fatigue tests is high enough to qualify as a "suddenly applied load" in the terminology of mechanics. Under these circumstances, the total stress in the fiber includes some dynamic wave form stress as well as the known static stress.

The combination will momentarily be somewhat less than twice the static loading. If this exists, we should not be surprised to find a much larger proportion of fibers failing on load application than would be indicated by the static load alone.

III. CURRENT EXPERIMENTAL WORK

Static Fatigue Tests at Liquid Nitrogen Temperature

The first several static fatigue tests at low temperature were concluded prematurely because of ice accumulation in locations which made the operation of the timer circuits undependable. Although five separate runs were started and valid data on percentage of initial failures obtained, all were terminated before the presence or absence of static fatigue was conclusively established. It appeared necessary to isolate the equipment completely from the atmospheric humidity, and this was done by building a plastic enclosure around the equipment. An electric dehumidifier is operated continuously inside the enclosure. During a run at low temperature, the input of liquid nitrogen, as it vaporizes, maintains a slight positive pressure inside the enclosure which helps keep outside moisture-laden air from entering. Performance

of the equipment with the new modifications has been much improved and the low temperature static fatigue tests should be concluded in October 1964.

In some of the early runs at low temperature when it became impossible to continue the test at -196°C for one reason or another, the loads were allowed to remain on the fibers as warm-up began. Fibers which had not failed under static load at -196°C were observed to fail as the temperature was allowed to rise slowly. The failures occurred at various temperatures in the range from -145°C to -75°C . It would seem, again, that a temperature-sensitive failure mechanism is indicated, and that the rate is appreciable even at quite low temperatures. The process is probably not completely absent at liquid nitrogen temperature, and we should expect delayed failure to occur if we were willing to continue the experiments long enough. It might be revealing to load fibers at liquid nitrogen temperature, then raise the temperature to various intermediate levels and hold it constant to observe delayed failure times as a function of temperature and load. Unfortunately, such an extended program is not within the scope of the present project.

IV. DISCUSSION

The explanation of the causes for glass fibers being weaker in practice than they are in theory is not yet completely clear. For many years there have been attempts to deduce the mechanisms of failure from experimental observations. In most cases a single process was envisioned to account for all failures in a variety of environments. Indeed, a particular chemical reaction, or series of reactions, between atmospheric water vapor and components of the glass has been most often suspected as being responsible for degradation of the molecular structure and consequent loss of strength. Since surface cracks can be shown to exist on bulk glass by appropriate etching techniques, it has been common practice to assume that similar flaws exist on the surface of the small (less than 10 micron diameter) glass fibers such as are presently being used in reinforced plastics structural applications. Such cracks can be shown mathematically to be capable of propagating under a stress which, if uniformly distributed over the entire macroscopic cross section, would not cause failure. The function of external water vapor in the failure mechanism is to increase the degree of stress concentration at the tip of the crack by reacting with components of the glass at a rate which depends upon the stress conditions at and near the reaction sites. It is presumed that mechanical

stress alters the normal, unstressed reaction rate by means of increased diffusion of the reacting species or reaction products.

There does seem to be reason to wonder if all of the activity leading to failure in virgin glass fibers must necessarily be restricted to the fiber surface. For example, experiments in which fiber surfaces have been intentionally dried have still shown evidence of delayed failure. Thus, in preliminary experiments with pure silica fibers immersed in a solution of aluminum hydride in ether and tested in bending, Hillig and Charles at the General Electric Research Laboratory found that the maximum strain was still time-dependent, although much higher than the normal atmospheric values. One is tempted to explain this by incomplete removal of moisture from the surface, but there is the possibility that either the reactants or the flaws or both can also be internal. Of course, reactions leading to degradation would be subject to retardation at low temperatures regardless of whether the activity were external or internal.

In our tests with E-glass fibers under rapid load application, we find that some fibers in a group break immediately at room temperature under a nominal load which does not break any fibers at liquid nitrogen temperature, yet other fibers in the same group fail only after bearing the static load for one week or more at room temperature. If stress corrosion can be

blamed for the difference in immediate failures at the two temperatures, then there would appear to be some flaws which are extremely susceptible and which require only a fraction of a second of reaction time at room temperature to propagate and cause failure, and other flaws which require up to one million times as long. These flaws which cause immediate failure at room temperature but not at liquid nitrogen temperature cannot depend upon extensive diffusion of reactants to the crack tip area and hence are most likely at the surface where water molecules are in plentiful supply. However, internal flaws would require considerably more time for reactants to diffuse through the glass structure from sites either within the glass or from the outside.

The suspicion of internal as well as external flaws is easily aroused when one observes the glass marbles which go into the melt from which the fibers are drawn. There are numerous bubbles in every marble, and even after a substantial period of fining at or above the drawing temperature, one still finds bubbles in the glass which drops from the orifice just preceding the fiber drawing. By the time the glass flow is attenuated into fibers of the usual diameter, it is not possible to detect any bubbles or other flaws even with excellent microscopes. Nevertheless, we are faced with great differences in

behavior under stress for various sections of a given fiber which can only logically be explained by a wide distribution of flaw severities.

We are hopeful that static fatigue tensile tests in desiccants, together with the present series at low temperature, will shed more light on the flaw picture.

V. FUTURE PLANS

In tests completed thus far at liquid nitrogen temperature, the static stress levels have been 500,000 psi and above, where the percentage of immediate failures is quite large. Future tests at -196°C will be principally at lower stress levels. As reported in our second bi-monthly report, at room temperature there was a simple relationship between static stress level and percentage of immediate failures. It will be interesting to observe whether or not this same dependence exists at low temperatures.

When the tests at low temperature have been completed, work will begin on similar static fatigue experiments in the presence of highly efficient desiccants at room temperature.